

5.6.2000

VIITTEEMME:  
OUR REFERENCE:

VUO 2 PCT

Dear Sirs,

Re: International Patent Application No. PCT/FI99/00359  
Metsä-Serla Oyj

With reference to the first written opinion of 28 March 2000, we respectfully submit the following:

Pages 17 to 19 are herewith replaced by the enclosed new pages 17 to 19.

Pending claims 1, 9 and 21 and 12 are replaced by the new claims bearing the same numbers.

The independent product claim 1 has been amended by incorporating a definition according to which the alkyl derivatives of cellulose are mainly alkali-soluble. This addition is based on the material appearing on page 6, line 10. The specific passage makes reference to CMC but, as mentioned on page 4, lines 23 to 29, the invention is illustrated with CMC and the described principles can be applied also to other similar cellulose derivatives. The crux of the embodiment referred to on page 6 at line 9 is the water-solubility in alkaline conditions, not that it would be comprised of CMC. Please also confer line 13 on page 5.

It is therefore obvious to a person skilled in the art that the disclosure of alkali solubility can be generalised to concern also other derivatives in addition to CMC.

Dependent claims 9 and 21 have been adapted to the new claim 1. The lower end value of the DP range is based on the disclosure on page 6 at line 31. The word “level” in claim 9 has been replaced with the proper term, viz. “degree”.

No new matter has been introduced.

Based on the clarified claim 1, the method according to the present invention for producing a modified fibrous product is now characterized in that

- the fibrous suspension is admixed at alkaline conditions with an alkyl derivative of cellulose which is soluble in water at mainly alkaline conditions and which is at least partially dissolved in water, and
- the derivative is allowed to attach to the fibrous raw-material before the drying of the fibrous material so that the attached cellulosic derivative cannot be removed by washing.

Nothing of this kind is disclosed in the references cited.

The closest prior art would appear to be represented by US Patent No. 5,354,424. Said publication discloses a method of coating paper with water soluble or water dispersable oligomers having a relatively low molecular weight. The oligomers are obtained by degrading polysaccharide derivatives, in particular starch and cellulose derivatives. The polymers can be degraded, e.g., enzymatically (cf. column 2, line 57 to column 3, line 34).

In the known method, paper is dipped into a treatment liquid containing oligomers or the treatment liquid can be added to a fibrous suspension (column 6, lines 12 to 64).

The reference therefore corresponds to the state of the art presented in the preamble of claim 1. Thus, in the known method, just as in the preamble, "the fibrous suspension is mixed with an alkyl derivative, which is at least partially dissolved in water".

In what respect does our invention then differ from the prior art of the reference ?

In our opinion there are at least three differences:

First, according to the reference, the alkyl derivative used is not water soluble "mainly in alkaline conditions". For CMC this requires that the DS must be below 0.5 and the DP preferably in the range of 600 to 5,000. Both of these prerequisites must be in force simultaneously.

In the reference no attention is paid to the degree of substitution of the CMC. The DS of the degraded polymer derivative should, according to the reference, be 0.1 to 3.0 (column 4, lines 39 to 41), in other words the DS can have any value. It is essential in the reference that, by means of the degradation procedure, an oligomer mixture is provided having a low polymerisation degree (DP about 3 to 500, preferably 3 to 300, in particular 5 to 50; cf. column 2, lines 57 to 63; column 5, lines 52 to 58). The claims are restricted to 5 to 50. As a result of these low DP values, the derivatives are water-soluble.

Secondly, the reference does not indicate at which pH the derivative is added to the stock. According to the present invention the cellulosic derivative is mixed at alkaline conditions with

the fibrous stock. This comprises, e.g., that the derivative is dissolved in an alkaline aqueous solution at a desired point of time and that the derivative is attached to the fibres from that solution.

Thirdly, as a result of the above differences, the short contacting times (5 and 10 minutes, respectively) are not sufficient to attach the cellulosic derivative so that it cannot be removed by washing. Since the derivatives are water soluble they are also easily washed away from the fibres. The invention uses generally longer contacting times, but also in the case when the time periods are in the same range of 5 to 10 minutes, the bonding is more complete because the derivatives are alkali soluble.

We have in our own experiments found that when CMC having a  $DP_v$  of 170 and DS of 0.57 (Finnfix BW-2) is used, no attachment of the derivative to the fibres can be achieved from a pure aqueous phase. By adding 1 M sodium acetate a partial attachment is obtained but the degree of sorption is, however, too low (about 20 % of the CMC). By contrast CMC grades having a higher DP and lower DS [e.g. Nymcel ZSB 10 ( $DP_v$  750, DS 0.20) and Nymcel ZSB 16 ( $DP_v$  700, DS 0.32)] attach well whereby at least 40 wt-%, preferably even 60 to 95 wt-% of the CMC is sorbed to the fibres. In our tests, with a DS of 0.2 the sorption was 94 %, with DS 0.32, the sorption was 69 % and even with DS 0.43, 38 % was sorbed.

The reference discloses that the properties of water-removal of the treated pulps and the mechanical properties of the pulp product are improved. This is also the case for the present invention, however with the difference that the improvement can be obtained with a much smaller amount of cellulosic derivative. In the reference, a 10 % CMC solution is used (Example 5) or slightly more than 5 % of the fibres (Example 7), whereas the amounts used in the present invention are smaller (1 % of the fibres in Example 1).

In spite of the smaller amounts used, the results are clearly better than those presented in the reference: according to Table 8 of the reference, the WRW value improves from 5 g to 5.3 g; for the present invention the WRW value becomes multifold in comparison to the reference (cf. Figure 2). The same conclusions can also be drawn regarding the strength properties (cf. Table 4 in the present application).

In summary we would like to point out that the present invention is based on the new concept of binding to the fibres a cellulose derivative which is alkali soluble, the bonding being carried out at conditions in which the derivative is so well bound that it cannot be washed away any more. The derivatives are attached to the surface of the fibres and even small amounts are sufficient to modify the properties of the fibres. Derivatives of small molecular size such as those used in the reference are water soluble and, as a result they are easily washed away. Obviously they also readily penetrate the cell wall of the fibres which increases the consumption of the derivative.

The other cited references represent a state of the art which is further away from the present invention.

Published PCT Application No. WO 96/3310 anticipates a preparation process of tissue paper wherein the fibrous stock is added CMC together with a bonding inhibitor (e.g. a quaternary ammonium compound) and cationic starch. The amount of CMC is 0.1 to 5 % and its DS is below 3, preferably in the range of 0.3 to 1.4. In the proposed DS range, the CMC grades are generally water soluble and, e.g. in the claims the use of water soluble CMC is specifically mentioned. No further details on the DP values are given in the reference.

EP Published Patent Application No. 0 273 075 concerns the preparation of paper with a capability of good water absorption. The fibres are admixed with a resin which absorbs water and a water soluble resin, e.g. CMC. Additives are added in an amount of even up to 80 % of the amount of the fibres (column 2, lines 45 to 54). The reference is completely silent about the afore-mentioned characterizing features.

With reference to the above stated favourable consideration of the attached new claims is herewith respectfully solicited.

Yours faithfully,  
Seppo Laine Oy

Christoffer Sundman

Encls.: new pages 17 to 19